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Determination and Separations of Gold in the Electrolytic Way.

THESIS

Presented to the Faculty of the Department of Philosophy of the University of Pennsylvania, in Partial Fulfillment of the Requirements for the Degree of Doctor of Philosophy,

BY
SARAH PLEIS MILLER, B.Sc.
PHILADELPHIA, PA.
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This investigation was undertaken at the suggestion of Professor Edgar F. Smith, and has been pursued under his direction. I wish to express my appreciation of his encouragement and unfailing kindness.

INTRODUCTION.

It has long been known that gold could be deposited completely from solutions containing it in the form of double cyanide, sulphaurate, and sulphocyanide, as well as in the presence of free phosphoric acid.

Smith's Electrochemical Analysis, 3rd Edition, gives an example which illustrates the conditions with which a good result may be obtained from the double cyanide; also those used to yield satisfactory results from sodium sulphide solution.

The purpose of this investigation was to ascertain the conditions under which gold could be satisfactorily deposited from its solution

(*a*) in potassium cyanide;

(*b*) in the presence of phosphoric acid;

(*c*) in sodium sulphide;

and to employ the data thus gathered for separations of the metal from various other metals.

GOLD FROM DOUBLE CYANIDE SOLUTION.

Determination of Gold.

The determination of the metal from its double cyanide solution first received consideration:

Smith's Electrochemical Analysis gives the following conditions: "The solution contained 0.1162 gram of metallic gold; to it were added 1.5 grams of potassium cyanide and 150 cc. of water. It was heated to 55° C. and electrolysed with a current of $N.D_{100} = 0.38$ ampere and 2.7–3.8 volts. The precipitation was complete in one and one-half hours; and the deposit weighed 0.1163 gram."

To 10 cc. of auric chloride were added 1.0 gram of potassium cyanide and 150 cc. of water. The gold precipitated was perfectly adherent and was deposited directly upon the sides of the platinum dish in connection with the cathode.

Results.

Gold present.	Potassium cyanide.	Dilution.	Current.	Volts.	Temperature.	Time.	Gold found.
Gram.	Gram.	cc.	$N.D_{100} =$		° C.		Gram.
0.1275	1.0	150	0.15–0.20 A	3.0–3.7	65	1 hour.	0.1275
0.1275	1.0	150	0.20–0.30 A	3.5–4.0	65	1 hour.	0.1277

With the view of separations that might possibly be made, a series of experiments were next carried out, employing the same electrolyte as prevailed in the above determinations, but using a current of low pressure and strength; in order to obtain conditions under which the lowest possible pressure and current strength would be applied and yet completely precipitate the gold from its solution.

Many trials were made with varying conditions. The temperature

best adapted to the deposition of the metal ranged between 55° C. and 65° C. In the cold the gold was not deposited so rapidly, and in fact, for the lowest amperage and voltage employed was not precipitated at all. A temperature above 75° C. also prevented to some extent the deposition of the gold.

A number of results obtained in this work are the following:

Number of Experiment.	Gold present.	Potassium cyanide.	Dilution.	Current.	Volts.	Temperature.	Time.	Gold found.
	Gram.	Gram.	cc.	$N.D_{100}$ =		° C.	Hours.	Gram.
1.....	0.1291	1.0	150	0.07–0.09 A	1.8–2.0	75	2½	0.1288
2.....	0.1291	1.0	150	0.04–0.06 A	1.8–2.0	68	2½	0.1293
3.....	0.1291	1.0	150	0.02–0.15 A	1.8–2.8	60	2	0.1294
4.....	0.1291	1.0	150	0.03–0.20 A	2.0–3.0	65	2½	0.1290
5.....	0.1291	1.0	150	0.11–0.04 A	2.5	61	2	0.1291
6.....	0.1291	1.0	150	0.03–0.02 A	1.8	70	2½	0.1290
7.....	0.1291	1.0	150	0.04–0.02 A	1.8	65	2½	0.1292

In experiments 1, 2 and 3, tabulated above, a current of $N.D_{100}$ = 0.015 A and voltage of 1.6 was allowed to act upon the solution for one hour; at the expiration of that time no gold had been precipitated.

It was also found that a current of $N.D_{100}$ = 0.02 A, and 1.8 volts, although sufficient to give a deposition of gold, failed to bring down the last traces. This was demonstrated by subjecting the solution to those conditions for three hours, and then raising the level of the liquid. After one hour, there being no further precipitation of metal, the liquid was siphoned off, the electrolyte replaced by water, the current interrupted and the deposit weighed after washing with hot water, alcohol and ether and drying in a desiccator. Instead of 0.1291 gram of gold present there was obtained (*a*) 0.1260 gram and (*b*) 0.1259 gram of metal.

As shown, however, in experiments 6 and 7, a current of 1.8 volts with amperage of 0.02–0.04 for one and one-half hours, and then increased so as to maintain a voltage of 1.8, allows of the complete precipitation of the gold.

The method of treating the depositions of metal as outlined in the above was adhered to in all experiments, unless otherwise stated. And in all cases (unless special treatment is cited) the gold was deposited directly upon the platinum; and the gold dissolved off, after weighing, by introducing very dilute potassium cyanide (0.5 gram in 150 cc. of water) into the dish, and connecting the latter with the anode of a battery yielding a very feeble current (0.2 A and 5.0 volts), using a copper wire in connection with the cathode. The time required to thus dissolve the gold from the dish was (for 0.1291 gram) one hour.

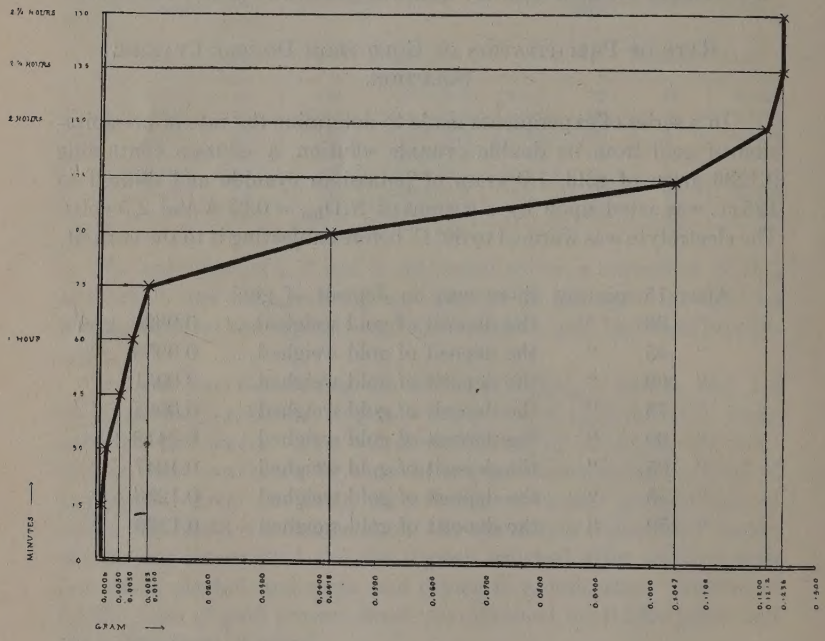
Rate of Precipitation of Gold from Double Cyanide Solution.

In a series of experiments made to determine the rate of precipitation of gold from its double cyanide solution, a solution containing 0.1236 gram of gold, 1.0 gram of potassium cyanide and diluted to 125 cc. was acted upon by a current of $N.D_{100} = 0.03$ A and 2.5 volts. The electrolyte was warmed to 50° C. before subjecting it to the current.

After	15	minutes	there was no deposit of gold.	
"	30	"	the deposit of gold weighed....	0.0006 gram.
"	45	"	the deposit of gold weighed....	0.0030 "
"	60	"	the deposit of gold weighed....	0.0051 "
"	75	"	the deposit of gold weighed....	0.0083 "
"	90	"	the deposit of gold weighed....	0.0418 "
"	105	"	the deposit of gold weighed....	0.1047 "
"	135	"	the deposit of gold weighed....	0.1236 "
"	150	"	the deposit of gold weighed....	0.1236 "

CURVE SHOWING THE RATE OF PRECIPITATION OF GOLD FROM DOUBLE CYANIDE SOLUTION.

CURRENT USED = N D_{100} = 0.03 AMPERE AND 2.5 VOLTS



Separations in Double Cyanide.

In the American Chemical Journal, **13**, 417, Smith and Muhr report the separation of gold from palladium, platinum, zinc, cobalt, nickel and copper. The conditions for these same separations, in terms of ampere and volt, will be found in the Journal of the American Society, 21, 921, 922 and 923; and in Smith's Electrochemical Analysis, pages 175 and 176.

That there still exist gaps, in a possible list of metals associated with gold, where no separations are recorded, is evident.

Those conditions which allow of the separation of gold from copper, have been stated to serve equally well for the separation of gold from molybdenum, tungsten and osmium.

Among the metals, not as yet successfully separated from gold, may here be mentioned, iron, cadmium, silver, lead and mercury.

Gold from Iron.

The separation of silver from iron when present as a ferrous salt, is given on page 172 of Smith's Electrochemical Analysis, and it was thought, that under similar or nearly similar conditions, the separation of gold from iron might be accomplished.

Ferrous ammonium sulphate was taken in sufficient quantity to yield 0.1300 gram of metallic iron, and dissolved in 10 cc. of water, filtered immediately into 3.0 grams of potassium cyanide, warmed and quickly filtered—this procedure insured the presence of all of the iron in the ferrous state. To this dark brown solution were added 10 cc. of auric chloride, containing 0.1286 gram of metallic gold, and the whole diluted to 125 cc.

Results.

Number of Experiment.	Gold present.	Iron present.	Potassium cyanide.	Dilution.	Current.	Volts.	Temperature.	Time.	Gold found.
	Gram.	Gram.	Gram.	cc.	$N.D_{100}$ =		° C.	Hours.	Gram.
1.....	0.1286	0.1300	3.0	125	0.13–0.36 A	2.3–3.0	65	2½	0.1284
2.....	0.1286	0.1300	3.0	125	0.06–0.11 A	2.3–3.0	Cold	Night.	0.1285
3.....	0.1286	0.1300	3.0	125	0.17–0.30 A	2.5–3.0	64	3	0.1285
4.....	0.1286	0.1300	3.0	125	0.11–0.12 A	2.2–3.0	Cold	Night.	0.1284

By several experiments it was demonstrated that a current of $N.D_{100} = 0.05–0.11$ A, and 1.7–2.3 volts, other conditions as previously given, failed to completely precipitate the gold; 0.1261 gram being found, 0.1286 gram being present.

In all four experiments, cited above, *the gold was completely precipitated from the solutions, and was free from iron.*

Gold from Cadmium.

There is no separation of these two metals in alkaline cyanide solution recorded.

The following suggestion, which was thought to be advantageous, in the separation of mercury from cadmium, was carried out: Before subjecting the electrolyte to the action of the current, the solution was first heated to 60° or 65° C. and kept at that temperature throughout the determination.

Cadmium sulphate containing 0.1107 gram of cadmium, and 10 cc. of auric chloride (= 0.1200 gram of gold) and 1.0 gram of potassium cyanide with a total dilution of 125 cc. constituted the electrolyte.

Experiment 1.—A current of $N.D_{100} = 0.025$ A and 1.5 volts acted upon the solution for one hour; at the expiration of that time, there being no deposit of gold, the current was increased to 0.04 A and 1.6 V. After holding the current at the latter strength for one hour, there still being no deposit of metal, it was again raised—giving 0.085 A and 1.8 volts, when *cadmium was deposited carrying with it gold.*

Experiment 2.—A small platinum spiral anode had been used in the preceding experiment (5 cm. diameter). In this second trial a platinum wire with a single turn was connected with the anode. By this means a lower amperage was obtainable for a corresponding voltage—however, no deposition of metal occurred until the current registered $N.D_{100} = 0.035$ A and 1.8 volts—when *cadmium and gold were simultaneously precipitated.*

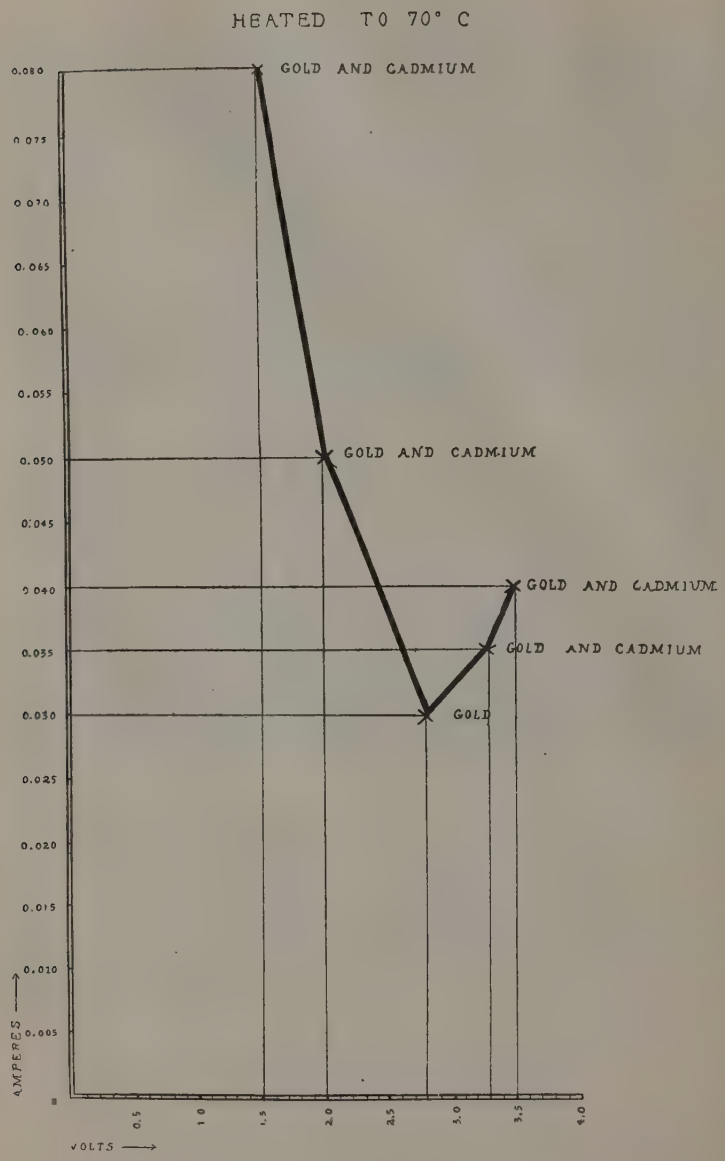
Experiment 3.—The solution remaining the same as in the two preceding experiments; in this third trial a straight platinum wire was connected with the anode in the hope of obtaining a current of sufficient pressure to precipitate the gold, but of current strength low enough to prevent the deposition of the cadmium. By this means a current of $N.D_{100} = 0.015$ ampere and 2.4 volts was obtained, but at the expiration of one hour there had been no precipitation of metal. The current was raised to 0.028 ampere and 2.5 volts; after one-half hour to 0.03 ampere and 2.8 volts. *A deposition of gold now occurred.* This deposit of gold did not seem to increase in quantity, so after one hour the level of the liquid was raised. A current of 0.035 ampere and 3.0 volts failed to give a further deposit of gold. On increasing it to 0.04 ampere and 3.5 volts, *cadmium was precipitated with the gold.*

Experiment 4.—Currents of $N.D_{100} = 0.03$ A and 1.6 V and 0.04 A and 1.8 V gave no precipitation of metal. On increasing the current to 0.05 A and 2.0 V, *cadmium and gold were deposited together.*

A determination was next made with the same electrolyte that prevailed in the four preceding experiments, but previous to the passing of the current the solution was not warmed, and was at no time above 50° C. In this case, *at first cadmium alone was deposited*, the current equalling 0.02 A and 1.5 volts. After one hour a film of *gold appeared on the cadmium deposit* and increased rapidly in amount (the current being, at the time of the appearance of the gold, 0.03 A and 1.7 volts).

CURVES BASED ON THE FACTS OF THE PRECEDING EXPERIMENTS.

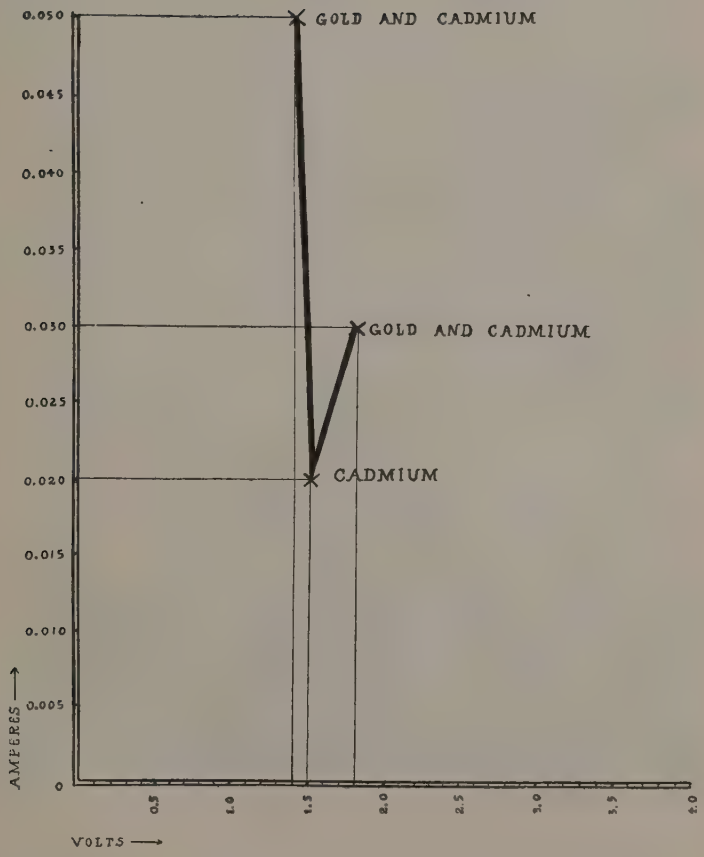
I.



CURVES BASED ON THE FACTS OF THE PRECEDING EXPERIMENTS.

II

IN THE COLD



Varying the amount of the potassium cyanide was tried. One and one-half grams, two grams, and two and one-half grams were used. In all of these trials the solutions were first heated to 60° C. A current of 0.035 ampere and 3.0 volts caused the *combined precipitation of cadmium and gold.*

Smith and Wallace, Journal of Analytical and Applied Chemistry (Jr. An. Ch.), **6**, 87, make mention that in the separation of gold from tungsten, when the tungstic acid was dissolved in caustic alkali and this solution then added to that of the gold, the gold was incompletely precipitated, although all the other conditions were favorable to its complete deposition.

This peculiar behavior of the gold led to the introduction of definite amounts of caustic alkali into cadmium solutions of known strength and containing known amounts of alkaline cyanide. For example, a current of 0.015 to 0.02 A, and 2.0 to 2.3 volts acting upon a cadmium sulphate solution (= 0.1107 gram of cadmium), 1.0 gram of potassium cyanide and 2.0 grams of caustic potash, diluted to 125 cc., and with a temperature of 70° C. failed to give any precipitation of cadmium.

A current of $N.D_{100} = 0.025$ A and 2.5 V with other conditions as prevailed in the preceding experiment, gave a complete deposition of cadmium.

It was hoped from the above observations to effect a separation of cadmium from gold by the retarding action of the potassium hydrate upon the gold or more probably upon the cadmium.

Experiment 5.—The separation of gold from cadmium was now tried. Cadmium was present as sulphate (= 0.1107 gram of metal), gold as auric chloride (= 0.1286 gram of gold in 10 cc.), to these were added 1.0 gram of potassium cyanide, and 2.0 grams of caustic potash. The whole diluted to 125 cc. and heated to 64° C. A current of 0.02 ampere and 1.6 volts was now allowed to act upon the solution, the outer circle of a large platinum spiral (of four turns) being in connection with the anode, this spiral was changed to a straight wire (3 cm. of its

length being immersed into the solution) which then gave a current of $N.D_{100}$ = 0.018 A and 2.2 V. Gradually increasing the current to 0.02 A and 2.4 V, gave no deposition of metal. With 0.02 A and 2.5 V *cadmium and gold were deposited together.*

Experiment 6.—In another trial, the current being $N.D_{100}$ = 0.017 ampere and 2.6 volts—other conditions as above—*cadmium and gold were both deposited.*

What may be gathered from the above?

In double cyanide solution, as may be observed from experiments 1, 2, 3 and 4, the *cadmium* with $N.D_{100}$ = 0.035 A and 1.8 – 3.5 *is invariably deposited with the gold,* which fact is true whether the solution be heated previously to the passing of the current, or whether this condition does not obtain. It is also true for the presence of varying amounts of potassium cyanide from 1.0 gram to 2.5 grams. But on adding 2.0 grams of caustic potash in presence 1.0 gram of potassium cyanide, cadmium is *not* precipitated when alone, with $N.D_{100}$ = 0.02 A and 2.3 volts, so that it was hoped that the separation could under these conditions be effected, but as exhibited in experiments 5 and 6, when the metals were associated, the lower current conditions there mentioned are useless—*both* metals being precipitated, *there being no separation of cadmium from gold.*

Gold from Silver.

In connection with the trials for the separation of gold from cadmium, mention has been made of the work of Smith and Wallace, Jr. An. Ch., **6,** 87, on the separation of gold from tungsten. That work also led its authors to experiment upon gold and silver. It was found by their researches, Jr. An. Ch., **6,** 87, that a "solution containing 0.1277 gram of gold, in the presence of 0.75 gram of potassium cyanide and acted upon by a current of 1.8 cc. electrolytic gas per minute, precipitated the metal fully in 16 hours. A similar solution, to which 2.0 grams of caustic potash had been added, when acted upon by a current of equal and even greater strength, did not

yield up any of its metal. Silver under conditions analogous to those described for gold, was fully deposited by a current generating 1.4 cc. of electrolytic gas per minute. . . . To 10 cc. silver solution (= 0.1090 gram of silver) were added 0.75 gram of potassium cyanide, 2.0 grams of potassium hydroxide, and 150 cc. of water. The current employed gave 1.4 cc. of electrolytic gas per minute. The precipitated silver weighed 0.1085 gram. Having thus found such a marked difference in the behavior of these two metals under the conditions mentioned, steps were taken to learn what the results would be if both metals were present together. It will suffice to say that their separation failed."

It was found by the authors of the above work, that in many cases, although the deposited metal was perfectly white in color, after weighing and solution in nitric acid, to be simply covering a layer of gold.

With these facts as a basis, solutions of gold and silver were separately subjected to the current:

A silver nitrate solution containing 0.1195 gram of silver to which had been added 1.0 gram of potassium cyanide and 2.0 grams of potassium hydrate diluted to 125 cc. was subjected to the current.

An auric chloride solution containing 0.1667 gram of gold and 1.0 gram of potassium cyanide and 2.0 grams of potassium hydroxide, 125 cc. dilution, was similarly treated.

Silver alone (0.1195 gram).	Gold alone (0.1667 gram).
Over night (cold).	*Over night (cold).*
$N.D_{100}$ = 0.009 to 0.015 ampere, and 1.6 to 1.8 volts.	$N.D_{100}$ = 0.01 ampere, and 1.6 volts.
= 0.1190 gram of silver found.	= No deposit.
Heated to 45° to 50° C.	*Heated to 45° to 50° C.*
$N.D_{100}$ = 0.01 to 0.02 ampere, and 1.5 to 1.75 volts.	$N.D_{100}$ = 0.015 ampere, and 1.6 volts.
= 0.1191 gram of silver found.	= No deposit.

With a current of $N.D_{100}$ = 0.025 to 0.03 ampere and 2.0 to 2.5 volts, 0.1579 gram of gold was deposited.

As will be observed from the results just cited, the voltage (1.8 *volts*) that *permits of the last traces of silver being deposited,* and that which would allow of *a precipitation of gold* (2.0 *volts*), are very close.

With these facts as a basis, the separation of silver from gold was tried.

The problem resolved itself into the effort to obtain, as far as possible, a high current strength with a very low pressure. In order to obtain such a relation between current strength and pressure the electrodes were varied in these respects: as to their size relation and their distance apart; the electrolyte was changed in regard to proportions of potassium cyanide and caustic potash present.

The auric chloride used contained 0.1200 gram of metallic gold in 10 cc.; 2.0 grams of potassium cyanide; the silver nitrate, containing 0.1199 gram of silver; 2.0 grams of caustic potash; and this solution was diluted to 125 cc.

Experiment 1.—At 5 o'clock in the evening the above solution was subjected to the action of the current—$N.D_{100}$ = 0.02 A and 1.5 volts. A perfectly white deposit began to appear almost immediately. The following morning the current registered 0.01 A and 1.4 V—and there was no indication of any metal on the platinum dish serving as cathode.

Experiment 2.—The same electrolyte as used for experiment 1. A temperature of 45° C. was maintained. The current $N.D_{100}$ = 0.02 A and 1.45 V at first caused precipitation of metal. After fifteen minutes the current fell to 0.015 A and 1.3 volts, and the deposit which had formed was redissolved. The current was raised to 0.025 A and 1.5 volts and deposition of metal began. After two hours the level of the liquid was raised. There seemed to be no further deposition of

metal; on raising the current to 0.03 ampere and 1.8 volts, however, more metal was deposited. In one-half hour the current was interrupted (after first siphoning off the electrolyte and replacing it with water) and the usual procedure of treating the deposit carried out—when the deposit appeared a uniformly light yellow in color. It weighed 0.1973 gram, the liquid was free from silver, hence 0.0774 gram approximately of gold had also been deposited.

Experiment 3.—The electrolyte was the same as used in the two preceding experiments. The current was held at 0.015 ampere and 1.6 volts for two hours, when the deposit, after washing with warm water, alcohol and ether, and drying, was weighed. There had been deposited 0.0935 gram of metallic silver (= 77% of silver present) absolutely free from gold.

Experiment 4.—The deposit which was obtained weighed 0.2003 gram, and had a pale yellow color. The current at first was $N.D_{100}$ = 0.02 A and 1.4 V. After two hours this was increased to 0.025 A and 1.7 V, and kept there for one-half hour. There was no indication of silver in the solution, so apparently 0.0804 gram of gold had also been deposited.

In another experiment with final results similar to those of experiments 2 and 4, the varying effects of temperature were demonstrated. In the cold a deposition of silver began with a current of 0.015 A and 1.45 V. A uniform coating, white in appearance, covered the dish. One hour later this had entirely disappeared. Heat was applied when the deposit again appeared. In a short time it again disappeared, the temperature registering at this time 78° C. On cooling the solution the coating again appeared and remained with a temperature ranging from 45° to 55° C.

With all of the preceding trials a platinum dish of 175 cc. capacity was used in connection with the cathode, and a large platinum spiral of 7 cm. diameter in connection with the anode.

Varying amounts of potassium cyanide and caustic potash were tried: 1.0 gram of potassium cyanide : 2.0 grams of caustic potash;

3.0 grams of potassium cyanide : 2.0 grams of caustic potash; and 4.0 grams of potassium cyanide : 2.0 grams of caustic potash.

In all the same results could be recorded—*failure to separate.* If a low enough current was used to yield silver free from gold, never more than 80% of the silver was deposited from its solution. When a current of sufficient strength to precipitate the last traces of silver was employed, varying amounts of gold were carried down with the silver.

Changes in the electrodes, and with each of these changes variations in the proportion of potassium cyanide to potassium hydrate, were next tried, as follows:

A platinum dish of 175 cc. capacity was made the anode, a platinum gauze (5 x 10 cm.) the cathode. In other experiments the platinum gauze served as the anode and the dish as the cathode.

Two plates of platinum (5 x 10 cm.) were used, the one, in connection with the anode, was rolled and fastened with platinum wire; the other surrounded it, being equally distant from it at all points; a beaker held the solution into which the electrodes were introduced.

When all of the silver was precipitated gold was invariably present. In some cases, the deposit was perfectly white in color, but when treated with nitric acid, the silver would be shown to have been covering a layer of gold. Oftener, however, the deposit was of a very pale yellow color, when in response to treatment with nitric acid the gold would be seen as a thin film covering the silver. In a few cases it seemed probable that the two metals had been simultaneously precipitated and were in consequence intimately combined in the deposit.

The expedient of first coating the platinum used in connection with the cathode received attention. Varying the electrolyte as to relative amounts of cyanide and caustic potash present; and also varying the electrodes as has been described above; in t his case also—but with no better results, *the silver and gold do not yield to separation.*

Gold from Mercury.

In the Zeitschrift für physikalische Chemie, **12,** 113, H. Freudenberg reports that the separation of mercury from gold is not practicable.

In the paper by Smith and Wallace in the Journal of Analytical and Applied Chemistry, **6**, 87, already referred to under the discussion of the separation of gold from cadmium, and gold from silver, the authors report having tried the separation of gold from mercury in cyanide solution containing known quantities of caustic alkali. The trials were fruitless and the investigations abandoned.

As the result of several experiments, it was plainly noticeable that the difference between the amperage and voltage where the mercury will be deposited and that which will permit of the precipitation of gold were too close to be available for purposes of separations. Experiments were instituted for both alkaline cyanide solutions, and alkaline cyanide solutions containing known quantities of potassium hydroxide; and these *two metals are yet not separated.*

IN PHOSPHATE SOLUTIONS.

Determination of Gold.

American Chemical Journal, **13**, 206. Smith reports "the precipitation of gold (0.1338 gram of gold) from a solution containing 20 cc. of disodium hydrogen phosphate (sp. gr. 1.038) and 3 cc. of phosphoric acid (sp. gr. 1.347). Total dilution = 160 cc. Current: 0.8 cc. OH gas per minute. The deposit weighed 0.1335 gram. The filtrate was found free from gold. The metallic deposit was quite adherent and compact. It was washed with hot water."

A solution containing 10 cc. of auric chloride (= 0.1286 gram metallic gold), 20 cc. of disodium hydrogen phosphate of 1.028 sp. gr., and 5 cc. of phosphoric acid of 1.35 sp. gr., was diluted to 125 cc., warmed to 65° C. before allowing the current to pass through; and electrolysed with a current of $N.D_{100} = 0.07$ A and 1.0 to 2.0 volts. After one and one-half hours the level of the liquid was raised and the current increased to $N.D_{100} = 0.12$ A and 2.5 V. There was no further deposition of metal.

The deposit was brown, and in washing, with the alcohol and ether, some of it was detached. The current used had evidently been too strong.

Other experiments followed, using a lower current; in these the deposits, although more compact and adherent, warm water only was used for washing, were not of bright yellow color. Dark specks appeared on the deposits. In weighing them, they were all found to be nearly 0.0008 gram too heavy.

The test made for phosphoric acid in the deposited gold failed to reveal its presence.

A solution of disodium hydrogen phosphate and free phosphoric acid was electrolysed, but gave no deposit.

Gently igniting the dish containing the deposit, by passing it several times over a small flame of a Bunsen burner, gave a bright yellow deposit of gold.

A deposit weighing ..... 0.0858 gram was thus treated,
and weighed.....0.0847 gram after the first heating,
" " .....0.0846 gram after the second heating,
" " .....0.0846 gram after the third heating.
There was present 0.0849 gram of gold.

The gentle ignition, as observed from the three weighings, was not sufficient to volatilise any of the gold, shown by the constant results obtained. The excess of weight of deposit being probably due to traces of organic matter in the disodium hydrogen phosphate or in the phosphoric acid.

RESULTS.

Number of Experiment.	Gold present.	Disodium hydrogen phosphate.	Phosphoric Acid.	Dilution.	Time.	Temperature.	Current.	Volts.	Gold found	
									Before Ignition.	After Ignition.
	Gram.	Sp. Gr. 1.028	Sp. Gr. 1.35	cc.	Hours.	° C.	$N.D_{100}$ =		Gram.	Gram
1.....	0.0849	20 cc.	5 cc.	125	2	60	0.02–0.015 A	1.0–1.8	0.0855	0 0847
2.....	0.0849	20 cc.	5 cc.	125	2	67	0.02 A	1.0–2.0	0.0857	0.0848
3.....	0.1196	20 cc.	5 cc.	125	3	60	0.03 A	1.0–2.0	0.1204	0.1197
4.....	0.1188	20 cc.	5 cc.	125	2	64	0.02–0.04 A	1.6–2.0	0.1196	0.1187
5.	0.1188	20 cc.	5 cc.	125	2	60	0.02–0.03 A	1.0–2.0	0.1192	0.1185

In all of these the deposits were perfectly adherent and free from phosphoric acid, and after heating were of bright yellow color.

Separations of Gold in Phosphate Solution.

Separations have been effected in this electrolyte, as will be found reported in the American Chemical Journal, **13**, 206, by Smith. That of gold from cadmium was found to be unsuccessful. The gold in separating either carrying down cadmium or separating in a spongy mass, this last occurrence being especially noticeable when the amounts of phosphoric acid were increased.

Gold from Cadmium.

Cadmium sulphate containing 0.1107 gram of cadmium was used. In every case the solution was previously heated to 50°–60° C. before allowing the current to act upon the solution. Disodium hydrogen phosphate (1.028 sp. gr.) and phosphoric acid (1.35 sp. gr.) were used in amounts of 40 cc. of the former and 10 cc. of the free acid; 30 cc. of the one and 8 cc. of free phosphoric acid; and 20 cc. of disodium phosphate and 5 cc. of free phosphoric acid. The results seemed to be equally satisfactory; 20 cc. of disodium hydrogen phosphate and 5 cc. of phosphoric acid were most frequently used.

No cadmium or phosphoric acid was found in the precipitated gold, and the deposits were perfectly compact and after heating bright in appearance. Warm water only was used in washing all deposits of gold from phosphate solution.

Results.

Gold present.	Cadmium present.	Dilution.	Temperature.	Time.	Current.	Volts.	Gold found after heating.
Gram.	Gram.	cc.	° C.	Hours.	$N.D_{100}$ =		Gram.
0.1188	0.1107	125	60	4	0.02–0.03 A	1.0–2.2	0.1190
0.1696	0.1107	125	65	4½	0 02 A	1.0–1.7	0.1697
0.1188	0.1107	125	55	5	0.02 A	1.1–2.0	0.1187
0.1448	0.1107	125	66	5	0.03 A	1.0–2.0	0.1450
0.1448	0.1107	125	66	4	0.02 A	1.0–1.8	0.1447

Gold from Iron.

Ferric ammonium sulphate was the salt used, containing 0.1100 gram of iron in 10 cc. Total dilution equalled 125 cc. The dish, with the deposited gold, was gently heated as described. The temperature was 52° C.; 40 cc. of disodium hydrogen phosphate (1.028 sp. gr.) and 10 cc. of free phosphoric acid (1.35 sp. gr.) and 0.1188 gram of gold were present.

The current acting upon the solution with a strength of $N.D_{100}$ = 0.03 to 0.06 ampere and 1.2 to 2.0 volts failed to precipitate the last traces of gold:

1. Gold found = 0.1163 gram.
   Gold present = 0.1188 gram.
2. Gold found = 0.1168 gram.
   Gold present = 0.1188 gram.
3. Gold found = 0.1155 gram.
   Gold present = 0.1188 gram.

The solution was made more dilute, and the current increased especially for the last half-hour. The results being then more satisfactory.

Results.

Number of Experiment.	Gold present.	Iron present.	Dilution	Time.	Temperature.	Current.	Volts.	Gold found after Ignition.
	Gram.	Gram.	cc.	Hours.	° C.	$N.D_{100}$ =		Gram.
1.....	0.1188	0.1100	150	6	63	0.02–0.16 A	1.0–3.6	0.1182
2.....	0.1188	0.1100	150	6	67	0.02–0.17 A	1.0–3.0	0.1190
3.....	0.1188	0.1100	150	5	62	0.02–0.08 A	1.0–2.7	0.1189
4.....	0.1188	0.1100	150	5	62	0.02–0.08 A	1.0–2.7	0.1189
5.....	0.1188	0.1100	150	5	62	0.02–0.08 A	1.0–2.5	0.1187
6.....	0.1188	0.1100	150	5	63	0.02–0.10 A	1.0–2.7	0.1185

In 1 and 2 the gold was not perfectly adherent. The current strength was lowered and in 3, 4 and 5 the *deposit was perfectly adherent*, of a *bright yellow color after heating*, and *was free from iron and phosphoric acid.*

Gold from Zinc.

In the American Chemical Journal, **13**, 206, Smith states that the separation of gold and zinc proceeded without the least difficulty. "A solution of 150 cc. volume contained, 0.1336 gram of gold, 0.1500 gram of zinc, 30 cc. of disodium phosphate (1.0358 sp. gr.), and 3 cc. phosphoric acid. It was electrolysed with a current giving 0.6 cc. of OH gas per minute. The gold deposit was compact and readily washed with hot water. It was dried over a warm iron plate. It weighed 0.1338 gram, zinc was not precipitated."

Zinc sulphate (= 0.1150 gram of zinc) and 10 cc. of auric chloride, 30 cc. of disodium hydrogen phosphate (1.028 sp. gr.), 6 cc. of phosphoric acid (1.35 sp. gr.) were diluted to 150 cc.

Results.

Gold present.	Zinc present	Time.	Temperature.	Current	Volts.	Gold found after heating.
Gram.	Gram.	Hours.	° C.	$N.D_{100}$ =		Gram.
0.1148	0.1150	3½	66	0.03 A	1.8-2.0	0.1151
0.1148	0.1150	3	61	0.03–0.04 A	1.0–2 0	0.1145
0.1117	0.1150	2	69	0.03–0.05 A	2.0–2.5	0.1113
0.1117	0.1150	2	69	0.03–0.05 A	2.0–2.5	0.1114
0.1117	0.1150	2	67	0.04 A	2.0–2.5	0.1115

Zinc was not found to be present in the precipitated metal.

Gold from Cobalt.

Reported in the same paper as the gold-zinc separation, American Chemical Journal, **13**, 206, and employing the same conditions as obtained in that separation, is the separation of gold from cobalt.

It was found after repeated experiments that the separation was effected more satisfactorily in rather dilute solutions. Accordingly a solution of auric chloride, 30 cc. of disodium hydrogen phosphate of sp. gr. 1.028, 6 cc. of phosphoric acid, sp. gr. 1.35, and cobalt nitrate containing 0.1200 gram of cobalt was diluted to 150 cc. to 200 cc.

RESULTS.

Gold present.	Cobalt present.	Time.	Temperature.	Current.	Volts.	Gold found after heating.
Gram.	Gram.	Hours.	° C.	$N.D_{100} =$		Gram.
0.1219	0.1200	2½	53	0.03–0.04 A	1.2–2.0	0.1216
0.1219	0.1200	4	55	0.04 A	1.2–2.3	0.1214
0.1219	0.1200	3	60	0.03–0.04 A	1.1–2.0	0.1217
0.1237	0.1200	2½	78	0.03 A	0.8–1.8	0.1235
0.1237	0.1200	3	58	0.04–0.08 A	1.1–2.2	0.1238
0.1237	0.1200	2	66	0.04 A	1.1–2.0	0.1236
0.1237	0.1200	2	62	0.03–0.06 A	1.1–2.0	0.1239
0.1237	0.1200	2	58	0.03–0.05 A	1.1–2.0	0.1235

Cobalt was not precipitated.

GOLD FROM NICKEL.

Since the separation of gold from cobalt had been effected it was natural to expect a like result for gold from nickel.

A solution of 150 cc. volume containing 0.1200 gram of nickel as nickelous nitrate, 40 cc. of disodium hydrogen phosphate of sp. gr. 1.028, 6 cc. of phosphoric acid, 1.35 sp. gr., 10 cc. of auric chloride containing 0.1236 gram of gold was electrolysed with the following results:

Gold present.	Nickel present.	Temperature.	Time.	Dilution	Current.	Volts.	Gold found after heating.
Gram.	Gram.	° C.	Hours.	cc.	$N.D_{100} =$		Gram.
0.1236	0.1200	55	4	150	0.04–0.07 A	0.8–2.4	0.1232
0.1236	0.1200	68	4	150	0.04–0.06 A	1.0–2.2	0.1236
0.1236	0.1200	62	3	175	0.03–0.06 A	1.0–2.0	0.1239
0.1236	0.1200	60	2	175	0.03–0.06 A	1.0–2.0	0.1233

The gold deposited showed no trace of nickel.

Gold from Copper.

The electrolyte consisted of copper sulphate (= 0.1200 gram of copper), 40 cc. of disodium hydrogen phosphate, 1.028 sp. gr., 10 cc. of phosphoric acid, 1.35 sp. gr., and 10 cc. of auric chloride (= 0.1188 gram of gold). Dilution, 125 cc.

A current of $N.D_{100} = 0.03$ A and 2.0 V acted on the above solution in the cold through the night. The deposit weighed 0.2047 gram; 0.1188 gram of gold being present. *Copper was present with the gold.*

In a second trial, using the same electrolyte as above, but a current of $N.D_{100} = 0.02$ ampere and 1.0 volt, with a temperature of 54° C., after three hours the deposit weighed 0.1112 gram of gold free from copper, 0.1188 gram of gold was present. This same current, however, failed to bring down the last traces of the gold from its solution.

In all precipitations of gold from acid phosphate solution, a platinum dish of 175 cc. to 200 cc. capacity was used in connection with the cathode, a platinum spiral with the anode. The depositions were washed only with cold and warm distilled water after siphoning off the liquid and replacing it with water. They were also heated gently either by passing the dish several times over a small flame of a Bunsen burner or by placing the dish on a warm iron plate.

IN SODIUM SULPHIDE SOLUTION

In Smith's Electrochemical Analysis it is stated "that the deposition of gold from sodium sulphide solution (sp. gr. 1.18) is just as satisfactory as that of gold from double cyanide."

The conditions given for the current are 0.1–0.2 ampere for a total dilution of about 125 cc. The precipitated metal was very adherent and of a bright yellow color.

Determination of Gold.

A solution of 150 cc., in which were 0.1276 gram of gold, 15 cc. of sodium sulphide (1.19 sp. gr.), heated to 61° C. was electrolysed with a current of 0.1 A and 0.1 – 0.2 A, and 2.4 to 3.0 volts. In two hours there was deposited (1) 0.1272 gram, and (2) 0.1277 gram of gold.

To the auric chloride were added 50 cc. of water, then the sodium sulphide and the solution warmed carefully until it became clear, and finally diluted to 150 cc. and electrolysed. The deposited gold is perfectly adherent, and of a shining yellow color, appearing like polished metal.

The sodium sulphide was prepared as follows: Sodium hydrate, precipitated from alcohol, in a solution of 1.25 sp. gr. Saturate one-half the quantity of the solution with hydrogen sulphide and filter quickly into the remaining one-half. Pass hydrogen sulphide through the entire solution to saturation, filter quickly. The yellow liquid is evaporated over a free flame quickly until a thin film forms on the surface. Allow the liquid to crystallize, redissolve, heat and repeat the crystallization and resolution of the crystals several times, then pour the hot solution (sp. gr. = 1.19) into glass-stoppered bottles, which are sealed.

Separations in Sodium Sulphide Solution.

The separation of gold from arsenic, molybdenum and tungsten, may be effected by observing those conditions given for the determination of gold from its sodium sulphide solution.

Separations of gold from mercury and tin have not been effected.

Gold from Mercury.

The solution electrolysed contained 10 cc. of mercuric chloride (= 0.1300 gram of mercury), 25 cc. of sodium sulphide (1.19 sp. gr.), 10 cc. of auric chloride (= 0.1276 gram of gold). Total dilution equalling 150 cc.

Several trials were made employing a current of $N.D_{100} = 0.015$ to 0.03 ampere and 1.1 to 1.7 volts. *In every case gold and mercury were precipitated together.*

Gold from Tin.

The many trials made to separate these two metals, in sodium sulphide solution, resulted in failures. *Tin being deposited with the gold.*